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APPLICATION OF EMULSIONS IN TRITIUM COUNTING WITH REFRIGERATED SYSTEMS

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Introduction

The use of emulsifiers in the low-level radioassay of tritiated aqueous samples by liquid scintillation counting at ambient temperature was investigated by this Laboratory and a new emulsifier for this purpose, Triton N-101, was introduced.(1)

Several investigations have been reported on the use of emulsions for liquid scintillation at refrigerated temperatures.(2,3) An optimum mixture reported with toluene-Triton X-100 incorporated 30 percent water with a tritium counting efficiency of 15.5 percent.

The objective of this investigation was to find an optimum emulsifier system that would incorporate 40 percent water and remain clear at temperatures below 10°C. The emulsifiers were evaluated on their ability to produce clear emulsion concentrates with the required amount of water and on the resultant counting efficiency.

Procedure and Results

Individual mixtures in polyethylene liquid scintillation counting vials were tested by refrigerating at 4°C and examined

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visually for periods up to 48 hours. Those mixtures clearing within 8 hours after the start of the refrigeration and remaining clear up to 48 hours were marked for further investigation.

Based on previous work with emulsions,[1,4] this investigation was limited to two primary solvents, toluene and p-xylene. The emulsifiers investigated were Triton X-100 and Triton N-101 because of their superior emulsifying properties with mixtures containing water and xylene or toluene. Triton X-45 was added to the mixtures as a hazepoint depressant in order to reduce the temperature range of the clear emulsion stage. All three emulsifiers are commercially available alkyl phenylpolyethylene glycol ethers, the Triton series being supplied from Rohm and Haas Co. Table 1 shows the results of a number of preliminary tests in which emulsifier-solvent proportions were varied.

It was decided, based on this test, to prepare a scintillation mixture containing fluors using the approximate concentrations of emulsifiers and toluene as shown in mixtures 5 and 7. The counting efficiencies of the two mixtures were found to be identical and mixture 5 was chosen for further examination.

The following mixture was prepared based on proportions from experiment 5.

Toluene	1000 ml
Triton N-101	296 ml
Triton X-45	94 ml
PPO	7.9 grams

Counting experiments were set up to determine the effect of secondary fluor, bis-MSB, on the counting efficiency of the above mixture when used with 40 percent water. The results are shown in Figure 1. Based on these results, 1.5 grams of bis-MSBwere used as the optimum amount for 1 liter of toluene mixed with 390 ml of emulsifier blend.

The final mixture used to incorporate 10 ml of tritiated water for each addition of 15 ml of scintillation solution was the following:

Toluene	1000 ml
Triton N-101	296 ml
Triton X-45	94 ml
PPO	7.9 grams
bis-MSB	1.5 grams

Liquid scintillation solutions based on the above composition were prepared and counted on a Packard TriCarb Liquid Scintillation Counter Model 3375. The sample compartment was operated at 8°C. It was noted that 70 to 100 minutes were required for samples to clear after being placed in the cooling compartment.

A total of 56 samples prepared with a tritiated water standard was counted. An average counting efficiency of 27.5 percent was obtained with an average background of 9.5 cpm. All samples were counted in polyethylene vials.

The minimum detectable activity (MDA) is expressed by the equation.[5]

$$M.D.A. = 3\sqrt{\frac{NBg}{tBg}}$$

where

NBg = count rate of background

tBg = counting time of background

The minimum sensitivity is expressed by the equation:[5]

$$M.S. = \frac{M.D.A.}{(eff.)(2.22\ dpm/pCi)(0.01\ liters)}$$

For 100-minute counting time, the minimum sensitivity averaged 152 pCi/liter of aqueous sample.

Some loss of toluene resulted after a one-week storage period and some separation of the emulsion occurred. A loss of approximately 20 percent of the original counting efficiency was noted after this length of time.[6]

Conclusions

An emulsion-type liquid scintillation solution was formulated for use between 0°C and 10°C.

The performance of the emulsifier system described above in counting aqueous samples containing tritium was better than than obtained with emulsifiers used at ambient temperature.

A limitation of the system is loss of toluene through the polyethylene counting vial. The instability of the emulsion caused by the loss of solvent limits the shelf life of prepared samples to less than one week. One possible remedy to this problem is the use of low-background glass vials of 25-ml capacity.

Mention of commercial products used in connection with work reported in this article does not constitute an endorsement by the Environmental Protection Agency.

Bibliography

1. Lieberman, Robert and Moghissi, A. A., Int. J. Appl. Radiat. Isotopes, 21 319 (1970).

2. Van der Laarse, J. D., Int. J. Appl. Radiat. Isotopes, 18 485(1967).

3. Williams, P. H., ibid. 19 377 (1968).

4. Benson, R. C., Analyt. Chem. 38 1353 (1966).

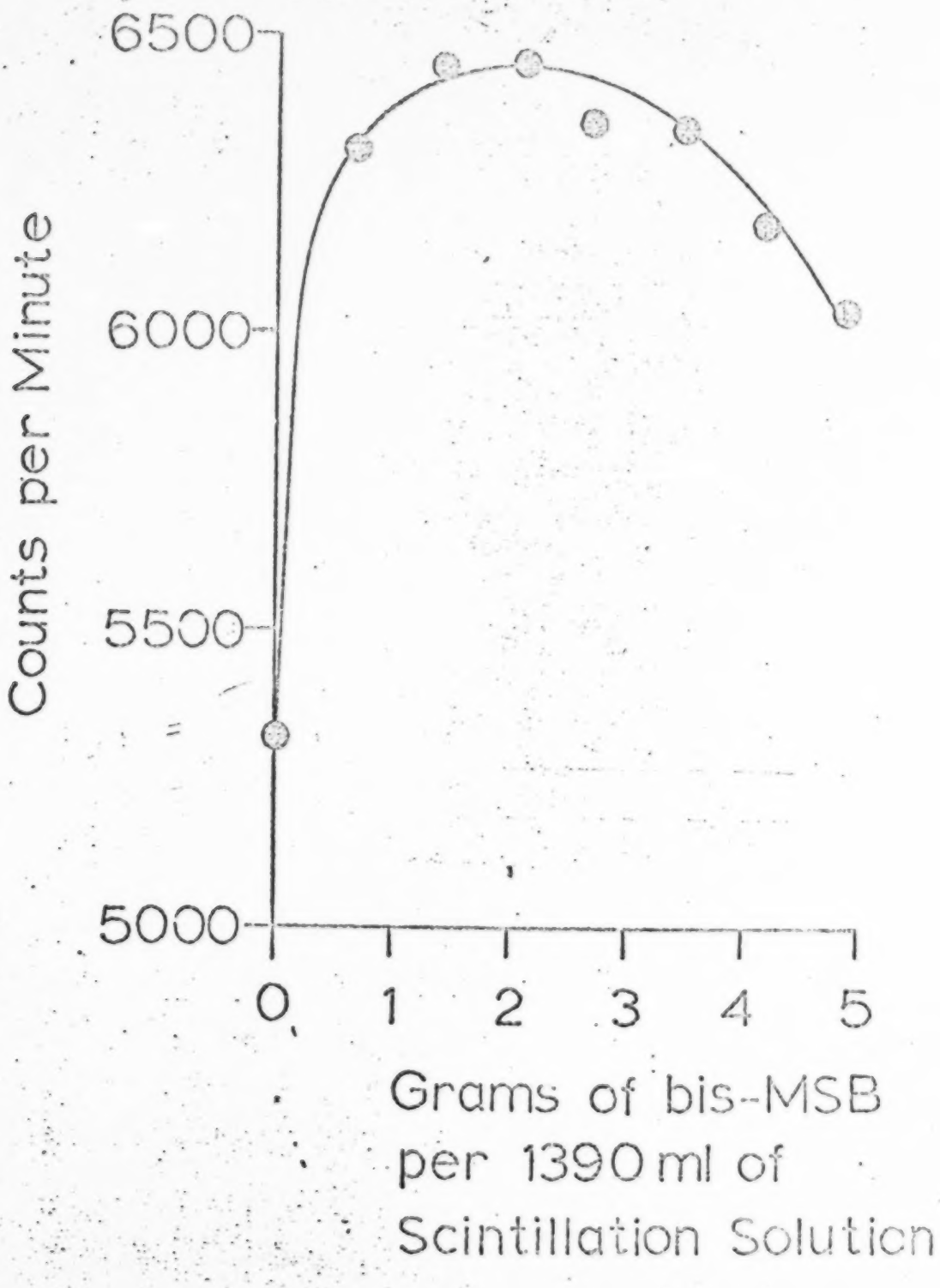
5. Training Publication No. 387n, U.S. Public Health Service.

6. Sedlet, Jacob, Argonne National Laboratory, private communication.

Table 1

RESULTS OF VISUAL INSPECTION OF EMULSIONS AT 4°C
CONTAINING 40% WATER

No.	Emulsifier, %	Solvent, %	Physical Appearance 8 hrs	48 hrs
1	Triton N-101 8.5 Triton X-100 8.5	toluene 43.0	Opaque	Opaque
2	Triton N-101 17.0	toluene 43.0	Opaque	Opaque
3	Triton N-101 8.5 Triton X-45 8.5	p-xylene 43.0	Opaque	Opaque
4	Triton N-101 8.5 TritonX-45 8.5	toluene 43.0	Opaque	Opaque
5	Triton N-101 13.0 Triton X-45 4.0	toluene 43.0	Clear	Clear
6	Triton N-101 13.0 Triton X-45 4.0	p-xylene 43.0	Opaque	Opaque
7	Triton N-101 12.0 Triton X-45 4.0	toluene 44.0	Clear	Clear


6500
6000
5500
5000
Counts per Minute
0
1
2
3
4
5
Grams of bis-MSB
per 1390 ml of
Scintillation Solution